STATE OF CALIFORNIA—BUSINESS, TRANSPORTATION AND HOUSING AGENCY

DEPARTMENT OF TRANSPORTATION
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METHOD OF TESTING CEMENT TREATED BASE AND CONCRETE FOR CALCIUM OXIDE

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part 2 of this method. It is the responsibility of whoever uses this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

SCOPE

This method describes the procedure by titration with potassium permanganate for the determination of calcium oxide and cement content in both cement treated base (CTB) and portland concrete cement.

PART 1. TEST METHOD

This test method is divided into the following parts:

1. Test Method
2. Safety and Health

A. APPARATUS

Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.

1. Hydrochloric acid: 36.5–38.0 % HCl by assay
2. Distilled water
3. Hydrogen peroxide: 30 %
4. Ammonium hydroxide: 28.0–30.0 % NH_3 by assay
5. Oxalic acid: 10 % solution
6. Ammonium oxalate: 5 % solution
7. Sulfuric acid: 95.0 to 98.0 %, H_2SO_4 by assay
8. Potassium permanganate: 0.1 N, standardized with National Institute of Standards and Technology (NIST) standard sodium oxalate or equivalent.
9. Beakers: 100 and 400 mL
10. Watch glasses: 65-mm diameter and 100-mm diameter
11. Hot plate
12. Volumetric pipettes: Class A: 5 and 20 mL
13. Buchner funnel: 90-mm inner diameter
14. Suction flask
15. Filter paper: 90-mm diameter, Munktell's OK or equivalent
16. Graduates: 5, 50 and 250 mL
17. Beaker tongs

18. 250-mL beaker with hot water

19. Stirring rods: glass

20. Funnel: Fluted, 60°, short stem, 50 to 55-mm inner diameter at top, borosilicate glass or polypropylene

21. Thermometer: 0-100°C

22. Buret: 0-5 mL with 0.1-mL gradations

B. PROCEDURE

1. Weigh into a 100-mL beaker 1 g of a dry sample which has been previously reduced to pass a No. 40 sieve. It will also be necessary to run a blank determination on a sample of the untreated base (UTB). Add 20 mL of HCl (1 + 1) and cover. Boil gently for exactly 5 minutes. Add 15 mL of boiling water. Filter with suction through a Munktell's OK paper.

2. Wash the residue with hot water and transfer the filtrate into a 400-mL beaker. Oxidize by heating with 6 to 8 drops of 30 % H_2O_2 and boil to remove the excess H_2O_2 . Adjust volume to 125 mL.

3. Heat to 70 to 80° C with stirring rod and slowly add NH_4OH (1 + 1) until the reddish precipitate of iron hydroxide just forms. (NOTE: Use a wash bottle and add the NH_4OH slowly with short squirts to allow large particle sizes to form). Add 8 to 10 mL of 10 % oxalic acid. About 4 to 5 mL of this should be sufficient to dissolve the precipitate if the previous step has been done correctly. The color should return to yellow green. With stirring rod, add 50 mL of 5 % ammonium oxalate and cover with the watch glass. Boil for five minutes and digest for 30 to 45 minutes at 60°C. The supernatant liquid should be clear. (The pH should be 3.5 to 4.5.)

4. Filter through a 90-mm OK paper into a 400-mL beaker and wash the precipitate ten times with hot water. The precipitate at this point should be pure white.

5. Place the filter paper in the original 400-mL beaker. Add 200 mL of water, and with stirring rod add slowly 5 mL of concentrated H_2SO_4 to the beaker. Using two glass-stirring rods shred the filter paper.

6. Heat to 90°C with stirring rod. Titrate with 0.1 N $KMnO_4$. Add the first 85 % $KMnO_4$ needed for titration rapidly. The end point is reached when the pink-red color persists for 30 seconds.

7. Calculation

$$\% \text{ CaO} = \frac{\text{mL } KMnO_4 \times N \times 0.028 \times 100}{\text{Wt. of sample, g.}}$$

$$\% \text{ Cement} = \frac{(\% \text{ CaO in CTB} - \% \text{ CaO in UTB}) \times 100}{\% \text{ CaO in brand of cement used} - \% \text{ CaO in UTB}}$$

8. Alternate Procedures

   Other methods that may be used are California Test 404 and ASTM Designation C-1084.

Part 2. SAFETY AND HEALTH

This method involves the handling of hazardous chemicals and samples, compressed and flammable gases, and sophisticated instruments, which can be dangerous.

Prior to handling testing or disposing of any waste materials, testers are required to read:

1. Caltrans Laboratory Safety Manual

2. Safety Precautions Applicable to 400 Series Test Methods in the California Test Methods

These sections pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc.

Observe good hygiene practices. Wash hands after handling samples and before eating, drinking or smoking.

Users of this method do so at their own risk.

REFERENCES:

ASTM Designation D-806

End of Test (California Test 403 contains 3 pages)